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(56) Documents Cited

GB 0732543 A EP 0281053 A2 US 5071864 A US 4775541 A US 4414231 A US 4205092 A

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(54) Method and kit for producing a fermented beverage

(57) The method comprises subjecting a fermented mixture to ion-exchange to remove unwanted flavours, such ion-exchange preferably being effected by use of a strong cationic resin and a strong anionic resin either as a mixture or sequentially. The beverage may be pre-treated with activated charcoal. A beverage of high alcohol content, viz. > 15% by volume, may thus be obtained without distillation. A kit for use in such method comprises yeast, yeast nutrients and ion-exchange material and, optionally, a flavouring.

DESCRIPTION

PREPARATION OF FERMENTED ALCOHOLIC BEVERAGES

The present invention relates to the preparation of fermented alcoholic beverages and more particularly, though not exclusively, to the production of home fermented high alcohol beverages.

Since in many countries high tax levels are levied on alcoholic beverages home brewing has become a popular past-time. One reason for this is that beverages made at home by fermentation are not subject to excise duty so long as they are not distilled and the alcoholic beverage is only for personal or family consumption and not for re-sale.

Thus, the home brewing of fermented alcoholic beverages such as, for example, beers, ciders, meads and wine have become increasingly popular. Such beverages typically have an alcoholic content of about 4 to 13% by volume.

To make alcoholic beverages with a higher alcohol content generally requires distillation. Thus, commercial alcohol for spirits and liquors is distilled to achieve the higher alcohol percentage required for these types of drinks. This is typically from around 25 to 40% alcohol by volume. Distillation allows selected fractions of distillate to be collected

according to the type of spirit required and the unwanted by-products and salts from the original fermentation are left behind. Activated charcoal may be used to remove unwanted flavours from the distilled alcohol. It can be dangerous for the amateur to distill alcohol to drink due mainly to the toxic and poisonous effects of the fusel oils if these are not removed. In some countries it is also illegal.

By using yeasts with a high alcohol tolerance in combination with larger proportions of specially formulated yeast nutrient salts high alcohol liquors have been produced without the need to distill the product thereby making them suitable for the homefermentation market. The high alcohol fermentation process does, however, produce a very salty/bitter tasting liquor. The unwanted flavours have to be masked using strong flavourings and sugar to make the fermented liquid palletable. Thus, the resulting fermented liquid is not suitable for making delicately flavoured drinks such as, for example, vodka, white rum, dark rum, whisky, brandy, sherry, gin and port.

The unpleasant tastes resulting from the production of high alcohol beverages is not, however, a problem in commercial production, since any unwanted flavours are left behind during the distillation

process. After distillation the distillate may, for example be diluted and/or flavoured or allowed to mature to taste.

However, for the home-fermentation market these unwanted flavours such as flavours resulting from an excess of salts and metabolic by-products are a serious problem since distillation is dangerous and probably illegal for the home brewer. Thus, to date, it has not been possible to produce delicately flavoured beverages of high alcohol content without distilling a fermented product.

An object of the present invention is to improve the taste of a fermented product without distilling it.

Another object of the invention is to produce a delicately flavoured alcohol beverage of high alcohol content without resorting to distillation.

By high alcohol content is meant greater than 15% alcohol by volume. Preferably the alcohol content is in the range of 15 to 25% alcohol by volume more preferably about 21% alcohol by volume.

According to the present invention there is provided a method of producing a fermented beverage which method comprises producing a fermented mixture and subjecting said fermented mixture to ion-exchange to remove unwanted flavours.

Preferably, the fermented beverage is a high alcohol beverage.

These unwanted flavours include amongst them excess yeast nutrient salts. These nutrient salts include, for example, nitrogen, potassium, magnesium, calcium and copper - usually in sulphate, phosphate and carbonate form, as well as meso-inositol and vitamins.

The addition of these yeast nutrients, acids and stabilizing chemicals are a necessary part of the high alcohol fermentation process yet their presence imparts a salty - chemical - bitter taste to the finished beverage. The actual taste depends upon the type and quantity of the chemicals present in the fermented liquid.

According to a further aspect of the invention there is provided a fermentation kit which in addition to the yeast and yeast nutrients comprises an ion-exchange material.

Preferably the fermentation kit is a high alcohol fermentation kit.

Preferably the ion-exchange material is a resin. More preferably the resin comprises a strong acid cation (H⁺) and a strong base anion (OH⁻), but alternatively a weak resin could be used.

The preferred resin is a mixture of a cationic

resin and an anionic resin. Indeed, where a mixture is used the mixture could comprise from 40% to 80% an anionic resin and from 60% to 20% a cationic resin; the percentages being by volume. Preferably the mixture contains between 65% to 55%, most preferably about 60% of a strong base anion (OH⁻) and between 45% to 35%, most preferably about 40% of a strong acid cation (H⁺). An example of a resin mixture comprising a strong acid cation (H⁺) and a strong base anion (OH⁻) is that sold by Rohm and Hass under the Trade Mark IMACHP5441A.

Examples of strong acid cation (H⁺) resins are D2990 and D2996 sold by Perolite International and examples of strong base anion (OH⁻) resins are D2991 and D2995 sold by Perolite International.

These or other cationic and anionic resins could be used sequentially.

The anionic and cationic resins can be of a variety of sizes. The smaller the particle size the faster the reaction with the salts. Typically the reaction will be conducted using resins with a particle size of between 0.3 mm to 1.2 mm.

The fermented liquor can be treated with the ionexchange material in a variety of ways.

The preferred method for amateur use is to add the mixed resin to the fermented liquor and to

agitate or stir the mixture so as to keep the resins in suspension for a time sufficient to remove the unwanted salts, typically from between 15 to 30 minutes.

After treatment the liquor should be tasted until it is confirmed free of a salty/bitter taste. If a salty/bitter taste is present more stiring and/or the addition of more resin may be required.

Since the resin mixture will have a higher wet density than the liquor it will precipitate quickly, usually within 1 to 5 minutes, when stiring is stopped thereby enabling the treated liquor to be decanted or syphoned off when the reaction is complete.

The quantity of resin required will vary in proportion to the quantity and according to the type of salts to be removed. As a guide, 20 grams of the mixed resin will remove about 1 gram of added nutrient salts.

A further 20 grams of the mixed resin, per litre of the liquor to be treated, should be added to cater for the removal of an average assumed quantity of natural salts contained in the original fermentation ingredients.

The preferred method for commercial use is to percolate the liquor through a bed of the mixed resin, in a column, at a rate of between 10 to 50 bed

volumes per hour. Preferably a conductivity meter is used to calculate the correct dose and confirm when desalinization is complete. After treatment the liquor will have a pH with a range of between 2.5 to 7, more preferably between pH 3 and 5.

Since a conductivity meter will not normally be available for the amateur it is preferred that the high alcohol kit contains a colour indicator in the resin to indicate when salt removal has taken place.

To further improve the taste of the product it is preferred to pre-treat the fermented liquor with activated charcoal prior to the ion-exchange step.

The choice of activated charcoal will vary according to the flavours and colours to be removed and also to any flavours or colours which are not to be removed.

For amateur use the preferred method is to add the activated charcoal to the liquor and stir or agitate the mixture to keep the charcoal in suspension in the liquor for a period sufficient to remove unwanted flavour and colour. This period is for a minimum of around 10 minutes and a maximum of 24 hours. Preferably this period is between 12 to 24 hours. Preferably the charcoal is in a powder form with a particle size of less than 0.1 mm. Using a small particle size speeds the absorption of

flavours. Preferably the dose of charcoal given is between 1 gram to 10 grams per litre of alcoholic liquor to be treated. The preferred dosage is 4.5 grams per litre. When all the colours and flavours have been absorbed silicic acid and chitosan is added to the mixture to floculate the charcoal so that the charcoal precipitates and allows the clear liquid to be syphoned or decanted off.

Preferably the home-fermentation kit is further provided with activated charcoal.

For commercial use the preferred method of pre-treatment is to filter the liquor through a bed of activated charcoal for a time sufficient to remove unwanted flavours and colours. The activated charcoal preferably has a particle size of from 0.4 mm to 2.0 mm.

The present invention will now be further described by way of example only with reference to the following examples:

EXAMPLE KIT

(To make 1 gallon of liquor)

Sachet of Nutrient mix - 20g

Sachet of Bentonite - 5g

Sachet of Yeast - 10g

Sachet of Potassium sorbate - 1g

Sachet of Liquid fining

e.g. silicic acid solution - 10g

Sachet of Refine

e.g. activated carbon - 20g

Sachet of Sodium metabisulphite - 0.5g

Sachet of Fining gel - 2g

Sachet of Superfine

e.g. ion exchange resin - 500g

comprising either a mixture of 60% of a strong base anion (OH⁻), and 40% of a strong acid cation (H⁺), such as IMACHP5441A from Rohm & Hass or a separate strong acid cationic (H⁺) resin such as, for example D2990 or D2996 from Perolite International and a separate strong base anionic (OH⁻) resin such as for example, D2991 or D2995 from Perolite International. Sachet of Flavour (optional)

N.B. Sugar - 1.6 kg bought separately

EXAMPLE OF PROCESS OF PRODUCING DELICATELY FLAVOURED "HIGH ALCOHOL" FERMENTATION LIQUOR

- 1. Pour 4 pints of cold water into a demi-john. Fit an airlock.
- 2. Pour 1 3/4 pints of cold water into a large saucepan and add 3½ lbs of white sugar (1.6 kg).

 Beat and stir the mixture to dissolve all the sugar no need to boil. Leave the solution to cool to 50°C (122°F) approximately, and pour 2 pints of this

solution from the saucepan into the demi-john. Pour the remainder into a 1 litre size lemonade type bottle, and seal with a cap. (This will be added to the demi-john in step 6, day 4). Check that the temperature of the liquor in the demi-john is less than 25°C (77°F); If not, wait for it to cool before continuing. Re-fit the airlock.

- 3. Empty the nutrient, 20g, into a clean, dry cup. Add the Bentonite, 5g, into the cup and mix together with a dry spoon. Add 2 to 3 oz, of warm water into the cup (30°C/86°F). Using a fork, whisk the contents of the cup thoroughly so that no lumps remain un-dissolved. Pour the contents of the cup into the demi-john, and pour back some of the liquid from the demi-john into the cup to rinse out all of the remaining un-dissolved nutrient into the demi-john. Repeat if necessary, mix well and re-fit the airlock.
- 4. Pour about ½ pint of cold water into a 1 pint jug. Using a thermometer add boiling water until the temperature reaches 40°C/104F. Pour some of this warm water into a cup, leave for 1 minute to warm the cup, then empty it down the sink. Pour 2 oz (50 ml) approximately of warm water (40°C/104°F) from the jug into the warm cup, add the yeast, 10g, stir to mix into warm water, cover and leave for 5 minutes then

mix thoroughly. Cover and leave the cup in a warm place for 20 minutes for the yeast to rise. Stir again and pour into a demi-john, rinsing out any remaining yeast from the cup into the demi-john with some of the liquor from the demi-john. Mix well and fit the airlock.

Leave for 4 days (Step 5)

5. Leave to ferment at a constant temperature of 20°C/68°F. Cooler or warmer temperatures varying between 18°C to 23°C (65°F to 74°F) are permissible but the fermentation in Step No: 7 may take a few days longer.

Day 4 (Step 6)

6. Pour sugar syrup from the lemonade bottle into the demi-john, rinse out any remaining sugar syrup from the bottle into the demi-john with about ½ pint of cool water (20°C/68°F). Top up demi-john to 6 pints 8 fl.oz with cool water (20°C/68°F) and mix well. Re-fit airlock.

Leave for 17 days (Step 7)

7. Leave to ferment at a constant temperature of 20°C/68°F for 17 days or until fermentation has finished. Cooler or warmer temperatures varying between 18°C to 23°C (65°F to 74°F) are permissible but the fermentation may take a few days longer.

Fermentation is complete when bubbles stop escaping through the air lock, the liquor starts to loose it's cloudy appearance and a thick sediment has formed at the bottom. A hydrometer reading of between 0.985 to 0.988 is observed. Wait until fermentation has finished before continuing.

DAY 21 (approximately) (step 8-14)

- 8. Pour liquid from the demi-john to splash into a clean dry bucket, taking care to pour slowly so that nearly all the liquid is decanted into the bucket and most of the unwanted sediment remains in the demi-john. Do not syphon. The pouring helps remove as much of the carbon dioxide gas as possible otherwise the liquid will not clear in instruction Step No: 18.
- 9. Empty the potassium sorbate, 1.0g, into the bucket and stir well.
- 10. Empty the liquid fining silicic acid solution- 10 g into the bucket and stir well.
- 11. Empty the re-fine activated carbon 20g into the bucket and stir well.
- 12. Rinse out the demi-john with clean water to remove sediment, and repeat several times if necessary.
- 13. Pour back the liquor from the bucket into the demi-john, rinsing out any remaining re-fine from the bucket into the demi-john with some of the liquor.

Re-fit airlock.

- 14. Pour 1½ fl. oz (40 ml) of cool water (about 20°C/68°F) into a clean 1 pint jug, add fining gel 2g and stir with a fork to mix in. Cover and leave for one hour, then mix well with a fork. Cover with cling film or similar and leave for between 12 to 24 hours. Then use as per instruction Step No: 17. Leave for 1 day (step 15)
- 15. Leave the demi-john for between 12 hours (minimum) to 24 hours (maximum) for the Refine to absorb unwanted fermentation flavours. During the same period, leave the covered jug of fining gel (mixed in Step No: 14) to dissolve to a smooth uniform texture.

DAY 22 (approximately) (Steps 16 and 17).

- 16. Empty the sodium metabisulphite 0.5g into the demi-john and mix well.
- 17. Pour about ½ pint of the liquor from the demijohn into the jug containing the fining gel (mixed the previous day in Step 14). Mix well with a fork. Add back into the demijohn, and agitate the demijohn to mix well.

Pour about 3/4 pint of the liquor from the demijohn into the jug to rinse out all the remaining gel into the demi-john. Now agitate the demi-john for about one minute to mix the liquor very thoroughly. Re-fit airlock.

- 18. Leave demi-john in a cool place until the liquor is perfectly clear and "brilliant". The liquor is normally clear with 24 hours, but it is recommended to leave it for 2 to 3 days (or sometimes longer) until the liquor is seen to be brilliant before continuing.
- 19. Syphon the clear liquor into a clean bucket.
- 20. Empty 500g of super-fine ion exchange resin (as per kit) into the bucket and mix with a plastic spoon to keep the super-fine particles in suspension and prevent them from settling to the bottom of the bucket for at least 20 minutes to remove unwanted salts, acids and minerals. One may either mix continuously for 20 minutes "all in one go", or one may prefer to mix for several shorter periods instead, taking a break between each mixing (e.g. 4 mixing periods of 5 minutes each = 20 minutes).
- 21. Leave the liquor undisturbed for 5 minutes to allow the super fine to settle, then syphon or decant the liquor into a clean demi-john. Transfer the superfine into a fine nylon sieve, straining bag or muslin cloth to strain out any remaining liquor from the super fine into the demi-john (use a large plastic funnel).

If making a vodka type liquor there is no extra

flavouring to add at this stage.

If making other spirit type liquors add appropriate flavourings into the demi-john and mix.

22. Finally, syphon or pour into bottles. Second hand spirit bottles with screw caps are recommended.

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-16-CLAIMS

- 1. A method of producing a fermented beverage which method comprises producing a fermented mixture and subjecting said fermented mixture to ion-exchange to remove unwanted flavours.
- 2. A method as claimed in claim 1, in which the fermented beverage is an alcohol beverage with an alcohol content of greater than 15% by volume.
- 3. A method as claimed in claim 2, in which the alcohol beverage has an alcohol content of from 15% to 25% by volume.
- 4. A method as claimed in claim 3, in which the alcohol beverage has an alcohol content of about 21% by volume.
- 5. A method as claimed in any of the preceding claims in which ion-exchange is conducted using a cationic resin and an anionic resin.
- 6. A method as claimed in claim 5, wherein the cationic resin and anionic resin are used as a mixture.
- 7. A method as claimed in claim 6, wherein the mixture comprises from 40% to 80%, the anionic resin and from 60% to 20% the cationic resin, the percentage being by volume.
- 8. A method as claimed in claim 7, wherein the mixture comprises from 55% to 65% the anionic resin

and from 35% to 45% the cationic resin, the percentage being by volume.

- 9. A method as claimed in claim 8, wherein the resin mixture comprises about 60% the anionic resin and about 40% the cationic resin, the percentage being by volume.
- 10. A method as claimed in claim 5, wherein the cationic resin and anionic resin are used sequentially.
- 11. A method as claimed in any of claims 5 to 10 wherein the cationic resin is a strong acid (H^+) resin and the anionic resin is a strong base (OH^-) resin.
- 12. A method as claimed in any of claims 5 to
 11, wherein the cationic resin and anionic resin have
 a particle size of from 0.3mm to 1.2mm.
- 13. A method as claimed in any of claims 5 to
 12, wherein the cationic and anionic resin are added
 to the fermented liquor; subjected to agitation or
 stirring for a time sufficient to remove any unwanted
 flavours; and the treated fermented liquor separated.
- 14. A method as claimed in claim 13, wherein the agitation or stirring is conducted for from 15 to 30 minutes.
- 15. A method as claimed in any of claims 5 to 12, wherein the fermented liquor is passed through a resin column.

- 16. A method as claimed in claim 15, wherein the fermented liquor is passed through the resin column at a rate of between 10 and 50 bed columns per hour.
- 17. A method as claimed in any of claims 5 to 16, wherein the resin is used in an amount of about 20g per gram of added nutrient salt.
- 18. A method as claimed in any of the preceding claims in which the fermented liquor is subjected to a pretreatment with activated charcoal before ion-exchange.
- 19. A method as claimed in claim 18, wherein the activated charcoal has a particle size of less than 0.1mm.
- 20. A method as claimed in any of the preceding claim, which further comprising adding a flavouring.
- 21. A method as claimed in claim 20 in which the flavouring imparts the flavour of one of the beverages selected from the group consisting of white rum, dark rum, whisky, brandy, sherry, gin and port.
- 22. A fermentation kit which in addition to yeast and yeast nutrients comprises an ion-exchange material.
- 23. A kit as claimed in claim 22 in which the fermented beverage is an alcohol beverage with an alcohol content of greater than 15% by volume.
 - 24. A kit as claimed in claim 23 in which the

alcohol beverage has an alcohol content of from 15% to 25% by volume.

- 25. A kit as claimed in claim 24 in which the alcohol beverage has an alcohol content of about 21% by volume.
- 26. A kit as claimed in any of claims 22 to 25 in which the ion-exchange material is a cationic resin and an anionic resin.
- 27. A kit as claimed in claim 26 wherein the cationic resin and anionic resin are present as a mixture.
- 28. A kit as claimed in claim 27 wherein the mixture comprises from 40% to 80%, the anionic resin and from 60% to 20% the cationic resin, the percentage being by volume.
- 29. A kit as claimed in claim 28, wherein the mixture comprises from 55% to 65% the anionic resin and from 35% to 45% the cationic resin, the percentage being by volume.
- 30. A kit as claimed in claim 29 wherein the resin mixture comprises about 60% the anionic resin and about 40% the cationic resin, the percentage being by volume.
- 31. A kit as claimed in any of claims 26 to 30 wherein the cationic resin is a strong acid (H^+) resin and the anionic resin is a strong base (OH^-) resin.

- 32. A kit as claimed in any of claims 26 to 30 wherein the cationic resin and anionic resin have a particle size of from 0.3mm to 1.2mm.
- 33. A kit as claimed in any of claims 22 to 32 which further includes activated charcoal.
- 34. A kit as claimed in claim 33 in which the activated charcoal has a particle size of less than 0.1mm.
- 35. A kit as claimed in any of claims 22 to 34 which further includes a flavouring.
- 36. A kit as claimed in claim 35, in which the flavouring imparts the flavour of one of the beverages selected from the group consisting of white rum, dark rum, whisky, brandy, sherry, gin and port.
- 37. A kit as claimed in any of claims 22 to 36 which further comprises a colour indicator to indicate when salt removal has taken place.
- 38. A method of producing a fermented beverage, substantially as hereinbefore described with reference to the example method.
- 39. A fermentation kit substantially as hereinbefore described with reference to the example kit.

Patents Act 1977 Examiner's report (The Search report	to the Comptroller under Section 17	Application number GB 9516007.3	
Relevant Technical	Fields	Search Examiner L V THOMAS	
(i) UK Cl (Ed.N)	C6E (EJCA	·	
(ii) Int Cl (Ed.6)	C12H 1/04	Date of completion of Search 7 NOVEMBER 1995	
Databases (see below) (i) UK Patent Office collections of GB, EP, WO and US patent specifications.		Documents considered relevant following a search in respect of Claims:- 1-39	
(ii) ONLINE: WPI			

Categories of documents

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Category	Identity	Relevant to claim(s)	
Х	GB 732543	(W & A GILBEY) see page 4, lines 37-80	1
X, Y	EP 0281053 A2	(HEUBLEIN INC) see page 2, line 49 - page 3 line 22 and page 3, lines 46-49	X:1 Y:13
X, Y	US 5071664	(BROWN) see column 1, line 63 - column 2, line 21 and column 2, lines 55-61	X: 1-5, 10, 11, 15 Y: 13
X	US 4775541	(BROWN ET AL) see column 4, lines 34-61 and column 5, lines 15-28	1, 5, 10, 11, 15, 22, 26, 31
x	US 4414231	(FICCA) see column 3, line 46 - column 4, line 32	1-4
X	US 4205092	(MATTICK ET AL) see column 2, lines 23-27 and 47-66	1, 5, 10

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

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(The Search report)

Continuation page

Category	Identity (Relevant to claim(s) X: 1 Y: 13	
X, Y	WPI Abstract Accession No 74-15086V/08 & SU 383458 A (MOLDAV FOOD IND) see abstract		
x	US 4156026	(GOGEL) see column 3, lines 8-22 and column 5, lines 31-33	1-5
X	US 3914442	(SERVADIO ET AL) see column 1, lines 31-42 and Claim 4	1, 2

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